Identifier:	Revision:	Effective Date:
ER-SOP-15.01	**0**	***4/27/00***

ER Document Catalog Number: **ER1999-0199**

Author: **Bart J. Vanden Plas**

environmental restoration project

A Department of Energy
Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Routine Validation of Volatile Organic Data

Los Alamos
NATIONAL LABORATORY
Los Alamos, New Mexico 87545

Routine Validation of Volatile Organic Data

Table of Contents

1.0 PURPOSE 4
2.0 TRAINING 4
3.0 DEFINITIONS 5
4.0 BACKGROUND AND PRECAUTIONS 8
5.0 EQUIPMENT 8
6.0 PROCEDURE 9
6.1 Prepare for Data Validation 9
6.2 Verify Instrument Performance Check 10
6.3 Verify Initial Calibration 11
6.4 Verify Continuing Calibration 12
6.5 Verify Method-Blank Results 12
6.6 Verify Internal Standards 16
6.7 Verify Surrogate Recoveries 22
6.8 Verify Holding Time 28
6.9 Verify Tentatively Identified Compounds 28
6.10 Complete the Data Validation Cover Sheet 29
6.11 Assemble the Validation Data Record Package 29
6.12 Submit the Validation Data Record Package 30
7.0 REFERENCES 30
8.0 RECORDS 30
9.0 ATTACHMENTS 30

List of Figures and Tables

Figure 6.5-1. LANL qualifier flags and reason codes for noncompliant method blanks. 15
Figure 6.6-1. LANL qualifier flags and reason codes for noncompliant IS compounds. 21
Figure 6.7-1. LANL qualifier flags and reason codes for noncompliant surrogate compounds. 27

Table 6.6-1. ER Project-Approved VOC Internal Standards 16
Table 6.7-1. VOC Surrogates and Recovery Acceptance Ranges 22
Table 6.8-1. Holding Time Acceptance Criteria. 28

List of Acronyms and Abbreviations

BFB	bromofluorobenzene
BSS	background subtracted spectra
CCV	continuing calibration verification
CLP	Contract Laboratory Program
COC	chain of custody
EPA	US Environmental Protection Agency
EQL	estimated quantitation limit
ER	environmental restoration
GPC	gel permeation chromatography
IS	internal standard
LAL	lower acceptance limit
LANL	Los Alamos National Laboratory
MDL	method detection limit
n/a	not applicable
%R	percent recovery
QC	quality control
RIC	restructured ion chromatogram
RN	request number
RPF	Records Processing Facility
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work
TIC	tentatively identified compound
UAL	upper acceptance limit
VOC	volatile organic compound

Routine Validation of Volatile Organic Data

NOTE: Environmental Restoration (ER) Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/documents/Procedures/sops.htm. However, it is their responsibility to ensure that they are trained to and use the current version of this procedure. Contact the author if the text is unclear.

1.0 PURPOSE

This standard operating procedure (SOP) represents the minimum standard for evaluating routine volatile organic compound (VOC) analytical data. These data can be generated for the Los Alamos National Laboratory (LANL) ER Project using SW-846 Method 8260, the comparable Contract Laboratory Program (CLP) methods under the current statement of work (SOW) for analytical services (LANL 1995), or EPA Method 624 for surface water analyses. The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used as a point of departure for developing focused data validation requirements specific to a particular data use.

Note: Implementation of this procedure will result in a tabulation of data compliances and noncompliances identified relative to expectations for data quality based on national guidelines for data review (EPA 1994). Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as *general indicators* of data quality and should not be construed as a definitive identification of data usability.

Note: Implementation of this procedure may be followed by a more focused and data use-specific evaluation of data, especially if implementation of this SOP indicates that the data may contain technical deficiencies.

2.0 TRAINING

All data validators implementing this SOP shall possess a minimum of a bachelors degree in chemistry and two years of experience in generating analytical data in an environmental analytical laboratory, or two years' data validation experience. New validators shall work under the direct supervision of an experienced ER Project validator. The work of new validators shall be reviewed and signed by an experienced ER Project validator until ten data record packages for each analytical suite have been satisfactorily validated. ER Project validators shall have demonstrated familiarity with the US Environmental Protection Agency (EPA) national functional guidelines for data review. All data validators must document

that they have read and understand this SOP and completed all applicable training assignments in accordance with QP-2.2.

3.0 DEFINITIONS

3.1 *Area count* — Integrated area under a chromatographic peak. The area count is proportional to the amount of compound present in the aliquot injected into the chromatograph.

3.2 *Continuing calibration verification (CCV)* — Combination of calibration blank and check standards used to determine if the method response to analyte concentration is within acceptable bounds relative to the initial calibration. A CCV is performed every 12 hrs of operation and establishes the 12-hr relative response factors on which quantitations are based, thus verifying an instrument's satisfactory performance on a day-to-day basis. The continuing calibration 12-hr period assumes that the gas chromatograph/mass spectrometer has not been shut down since its initial calibration.

3.3 *Data validator* — Person who has met the minimum standards of training established in Section 2.0 and who implements this SOP on behalf of the ER Project.

3.4 *Detect* — Sample result greater than the method detection limit (MDL) reported by the laboratory. The laboratory reports the concentration of the analyte in the sample.

3.5 *Estimated quantitation limit (EQL)* — Lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The low point on a calibration curve should reflect this quantitation limit. The EQL is not used to establish detection status. See the SOW for analytical services (RFP No. 9-XS1-Q4257) for a more complete definition.

3.6 *Holding time* — Maximum time between sample collection and sample preparation and/or analysis that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed storage conditions; deviations in storage conditions may affect holding times. Appropriate storage conditions for samples of various matrices scheduled for selected analyses may be found in the current LANL-ER-SOP-01.02, the applicable analytical method, and the current ER Project SOW for analytical services.

3.7 *Initial calibration* — Process used to establish the relationship between instrument response and analyte concentration at several analyte-concentration values to demonstrate that an instrument is capable of acceptable analytical performance. The initial calibration for VOC analyses is

performed at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not satisfied.

3.8 *Instrument performance check* — Analysis of a chemical of known relative mass abundances that indicates how well a mass spectrometer is calibrated.

3.9 *Internal standard (IS)* — Chemical compound added to every blank, sample, and standard extract at a known concentration that is used to (1) compensate for analyte concentration changes that might occur during storage of the extract and (2) compensate for quantitation variations that can occur during analysis. ISs are used as the basis for quantitating target analytes.

3.10 *Laboratory duplicate sample* — The portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is expected to be equally representative of the original material. Duplicate analyses also are performed to generate data, to determine the long-term precision of an analytical method on various matrices.

3.11 *Laboratory qualifier (or laboratory flag)* — Codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA CLP guidelines.

3.12 *LANL data validation qualifiers* — The data qualifiers defined by LANL and used in the ER Project baseline-validation process. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP (ER-SOPs 15.01–15.06).

3.13 *LANL data validation reason codes* — The codes applied to the sample data by data validators who are independent of the contract laboratory which performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier with some description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP (ER-SOPs 15.01–15.06).

3.14 *Lower acceptance limit (LAL)* — Lowest limit that is acceptable, based on the quality control (QC) criteria for a specific QC sample for a specific method. Any results lower than the LAL are qualified following this routine validation procedure.

3.15 *Matrix spike* — An aliquot of sample spiked with a known concentration of target analyte(s). Matrix spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. The spiking typically occurs before sample preparation and analysis.

3.16 *Matrix spike duplicate* — An intralaboratory duplicate sample spiked with a known amount of target analyte(s). Spiking occurs before sample preparation and analysis.

3.17 *Method blank* — Analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. A method blank is used to assess the potential for sample contamination during preparation and analysis.

3.18 *Method detection limit* — Minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of samples of a given matrix type that contain the analyte after subjecting the sample to the usual preparation and analyses. The MDL is used to establish detection status.

3.19 *Nondetect* — Sample result that is less than the MDL. The laboratory reports nondetects as undetected at the EQL.

3.20 *Percent recovery (%R)* — Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.

3.21 *Request number (RN)* — An identifying number assigned by the ER Project to a group of samples that are submitted for analysis.

3.22 *Routine data* — Data generated using analytical methods that are identified as routine methods in the current ER Project SOW for analytical services.

3.23 *Routine data validation* — Process of reviewing analytical data relative to quantitative routine acceptance criteria. The objectives of routine data validation are to (1) estimate the data's technical quality relative to minimum national guidelines adopted by the ER Project, and (2) indicate to data users the technical data quality at a gross level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.

3.24 *Surrogate compound (surrogate)* — Organic chemical compound used in the analyses of organic target analytes that is similar in composition and behavior to target analytes but is not normally found in environmental samples. Surrogates are added to every blank, sample, and spike to evaluate the efficiency with which analytes are recovered during extraction and analysis.

3.25 *Target analyte* — An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.

3.26 *Tentatively identified compound (TIC)* — Chemical compound detected in a sample that is not a target analyte, IS, or surrogate compound. Up to 30 chromatographic peaks may be subject to mass spectral matching for identification as TICs.

3.27 *Upper acceptance limit (UAL)* — Highest limit that is acceptable, based on the QC criteria for a specific QC sample for a specific method. Any results greater than the UAL are qualified following this routine validation procedure.

4.0 BACKGROUND AND PRECAUTIONS

4.1 To protect the integrity of the data record package, the **data validator** must store and handle all data record packages under LANL ER Project chain of custody (COC) rules prescribed in ER-SOP-15.09.

4.2 Logic diagrams are included in this SOP to expedite the validation process Logic diagrams in this SOP do not include instructions for where to record validation results. Those instructions are incorporated in the text that corresponds to each logic diagram.

4.3 The VOC data validation checklists identify actions that must be taken, depending on whether a validation condition is true or false (Attachment D). Look at the top of each validation form to learn the required action.

4.4 This validation process requires that the **validator** record qualifier flags and reason codes on photocopies of the data summary results forms (Form I) in the hard copy data record packages. Contiguous lines of identical qualification on the photocopied Form I may be represented as the qualifier flag and reason code, followed by a vertical downward arrow to the end of the block of results that are qualified identically.

4.5 The VOC data validation checklist forms in Attachment D are examples of the forms the validator must use to validate data under this SOP. The forms may be reproduced in whole or in Part , as needed to complete the validation of a data record package.

5.0 EQUIPMENT

The **validator** may need the following equipment and supplies to implement this procedure:

5.1 current VOC data validation checklist forms (see Attachment D),

5.2 data record packages to be validated,

5.3 electronic calculator (optional),

5.4 photocopier, and

5.5 current ER Project SOW for analytical services.

6.0 PROCEDURE

Note: Deviations from SOPs are made in accordance with QP-4.2.

6.1 Prepare for Data Validation

6.1.1 The **validator** will begin by obtaining the required current versions of the VOC data validation checklist forms (see Attachment D) from the ER Project website (http://erinternal.lanl.gov/Quality/forms.htm).

6.1.2 Obtain from the Sample Management Office (SMO) of the Field Support Facility the data record package(s) that contain the sample data to be validated.

6.1.3 Prepare a data validation cover sheet (see Attachment C) by completing the top part of the appropriate form and placing a check or other mark adjacent to the analytical suites for which this validation is being performed.

Note: You may use a single cover sheet when validating multiple analytical suites under the same RN.

Note: Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure, including phone conversations with the analytical laboratory personnel concerning these deficiencies. Attach these sheets to the data validation cover sheet.

6.1.4 Verify that the following items are present in the data record package:

6.1.4.1 signed LANL COC record;

6.1.4.2 case narrative;

6.1.4.3 result forms (CLP Form I or equivalent) for each sample;

6.1.4.4 reconstructed ion chromatograms (RICs) for each sample;

6.1.4.5 RICs for standards;

6.1.4.6 raw and background subtracted spectra (BSS) of identified compounds;

6.1.4.7 quantitation reports;

6.1.4.8 QC forms (CLP 2A, 2B, 3A, 3B, 4A, 5A, 6A, 7A, 8A, or equivalent) for water and/or soils, as appropriate;

6.1.4.9 TIC forms (CLP Form V-TIC, or equivalent), which are required only if the ER Project requested TIC reports; and

6.1.4.10 mass spectra of TICs with three best library matches (required only if the ER Project requested TIC reports).

6.1.5 If the data record package does not contain all items listed in Sections 6.1.4.1 through 6.1.4.10, contact the analytical laboratory to obtain those materials.

6.1.5.1 If required documentation is missing from the data record package, and the package is less than six months old, contact the analytical laboratory and allow three business days for the laboratory to submit the required documentation.

6.1.5.2 If the analytical laboratory does not submit documentation within three business days, return the data record package to the SMO for contract-compliance action.

6.1.5.3 If the data record package is greater than 6 months old, allow 10 business days for the analytical laboratory to submit the required documentation before returning the data record package to the SMO.

6.1.6 Record the presence or absence ("Y" or "N") of each item, as appropriate, in the completeness section of the data validation cover sheet.

6.1.7 If the ER Project did not request TICs, record "n/a" (for "not analyzed") in blocks 9 and 10 of the completeness section of the data validation cover sheet.

6.1.8 In the data validation cover sheet completeness section, note any samples whose data are missing from the data record package.

6.1.9 Photocopy all analytical laboratory QC forms from the data record package.

6.1.10 Photocopy the case narrative from the data record package.

6.1.11 Photocopy the forms (Form I) that you will use during the validation process before completing the form.

Caution: Do not record data-validation qualifiers and reason codes on the original form (Form I).

Note: The **validator** must submit photocopies of the items listed in Sections 6.1.9 through 6.1.10 as attachments to the completed data validation checklists.

6.2 Verify Instrument Performance Check

6.2.1 If a bromofluorobenzene (BFB) instrument performance check *was completed* on the same date as, or within 12 hrs of, the corresponding sample analyses,

6.2.1.1 record "Y" in block 1a of the VOC data validation checklist, Part I;

6.2.1.2 record “n/a” in block 1c of the VOC data validation checklist, Part I; and

6.2.1.3 go to Section 6.3, Verify Initial Calibration.

6.2.2 If an instrument performance check (BFB analysis) *was not completed* on the same date as, or within 12 hrs of, the corresponding sample analyses,

6.2.2.1 record “N” in block 1a of the VOC data validation checklist, Part I;

6.2.2.2 circle “A, V16” in block 1b of the VOC data validation checklist, Part I;

6.2.2.3 record the qualifier flag and reason code combination “A, V16” next to the results for all affected samples, on Form I; and

6.2.2.4 record the time elapsed (to the nearest minute) between the instrument performance check and sample analyses in block 1c of the VOC data validation checklist, Part I.

6.3 Verify Initial Calibration

6.3.1 If the initial calibration *was completed* within 12 hrs of completing the instrument performance check,

6.3.1.1 record “Y” in block 2a of the VOC data validation checklist, Part I;

6.3.1.2 record “n/a” in block 2c of the VOC data validation checklist, Part I; and

6.3.1.3 go to Section 6.4, Verify Continuing Calibration.

6.3.2 If the initial calibration *was not completed* within 12 hrs of completing the instrument performance check,

6.3.2.1 record “N” in block 2a of the VOC data validation checklist, Part I;

6.3.2.2 circle “A, V16” in block 2b of the VOC data validation checklist, Part I;

6.3.2.3 record the qualifier flag and reason code combination “A, V16” next to the results for all affected samples, on Form I; and

6.3.2.4 record the time elapsed (to the nearest minute) between completion of the instrument performance check and completion of the initial calibration in block 2c of the VOC data validation checklist, Part I.

6.4 Verify Continuing Calibration

6.4.1 This validation check is not required if all samples were analyzed within 12 hrs of the initial calibration. In that case, record

6.4.1.1 "n/a" in blocks 3a and 3b of the VOC data validation checklist, Part I.

6.4.2 If a continuing calibration *was performed* on the same day as, or within 12 hrs of, the sample analyses,

6.4.2.1 record "Y" in block 3a of the VOC data validation checklist, Part I;

6.4.2.2 record "n/a" in block 3c of the VOC data validation checklist, Part I; and

6.4.2.3 go to Section 6.5, Verify Method-Blank Results.

6.4.3 If a continuing calibration *was not performed* on the same day as, or within 12 hrs of, the sample analyses, record

6.4.3.1 "N" in block 3a of the VOC data validation checklist, Part I and

6.4.3.2 circle "A, V16" in block 3b of the VOC data validation checklist, Part I;

6.4.3.3 record the qualifier flag and reason code combination "A, V16" next to the results for all affected samples, on Form I; and

6.4.3.4 the time elapsed (to the nearest minute) between completion of the continuing calibration and completion of analyses quantitated under the continuing calibration in block 3c of the VOC data validation checklist, Part I.

6.5 Verify Method-Blank Results

Note: The data validator must compare method-blank results to the contractually required EQLs.

Note: If additional validation forms are needed to record validation data for more than one blank, copy the appropriate forms and use the copies for the additional information.

Note: An analytical batch is any group of field samples and QC samples analyzed within 12 hrs of the BFB analysis.

6.5.1 If a method blank *was analyzed* for each sample matrix and/or analytical batch,

6.5.1.1 record “Y” in block 1a of the VOC data validation checklist, Part IIa;

6.5.1.2 record “n/a” in block 1c of the VOC data validation checklist, Part IIa; and

6.5.1.3 go to Section 6.5.3.

6.5.2 If a method blank *was not analyzed* for each sample matrix and/or analytical batch,

6.5.2.1 record “N” in block 1a of the VOC data validation checklist, Part IIa;

6.5.2.2 circle “A, V5a” in block 1b of the VOC data validation checklist, Part IIa;

6.5.2.3 record the qualifier flag and reason code combination “A, V5a” next to the results of all samples for which a method blank was not analyzed, on Form I; and

6.5.2.4 record which matrices and/or analytical batches did not include a method-blank analysis in block 1c of the VOC data validation checklist, Part IIa.

6.5.3 If *no* target analytes were detected in the method blank,

6.5.3.1 record “N” in block 2a of the VOC data validation checklist, Part IIb

6.5.3.2 record “n/a” in blocks 2c and 2d of the VOC data validation checklist, Part IIb; and

6.5.3.3 go to Section 6.6, Verifying Internal Standards.

6.5.4 If the concentration of acetone, 2-butanone, or methylene chloride in a sample is greater than the EQL and less than or equal to 10 times the concentration in the corresponding method blank,

6.5.4.1 record “Y” in block 2a of the VOC data validation checklist, Part IIb;

6.5.4.2 circle “U, V4” in block 2b of the VOC data validation checklist, Part IIb;

6.5.4.3 record the qualifier flag and reason code combination “U, V4” next to the result for each affected target analyte, on Form I;

6.5.4.4 record the samples that have been qualified “U, V4” and the analytes that were detected in the method blank in block 2c of the VOC data validation checklist, Part IIb; and

6.5.4.5 record the analyte names and their method-blank concentrations for acetone, 2-butanone, or methylene chloride detected in the method blank in block 2d of the VOC data validation checklist, Part IIb.

6.5.5 If the concentration in a sample of any analyte *other than* acetone, 2-butanone, or methylene chloride is greater than EQL and less than or equal to 5 times the concentration in the corresponding method blank,

6.5.5.1 record "Y" in block 2a of the VOC data validation checklist, Part IIb;

6.5.5.2 circle "U, V4" in block 2b of the VOC data validation checklist, Part IIb;

6.5.5.3 record the qualifier flag and reason code combination "U, V4" next to the result for each affected target analyte, on Form I

6.5.5.4 record the samples that have been qualified "U, V4" and the analytes that were detected in the method blank, in block 2c of the VOC data validation checklist, Part IIb; and

6.5.5.5 record the analyte names and their method-blank concentrations for any analyte other than acetone, 2-butanone, or methylene chloride detected in the blank, in block 2d of the VOC data validation checklist, Part IIb.

6.5.6 Use the logic diagram in Figure 6.5-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant method blanks.

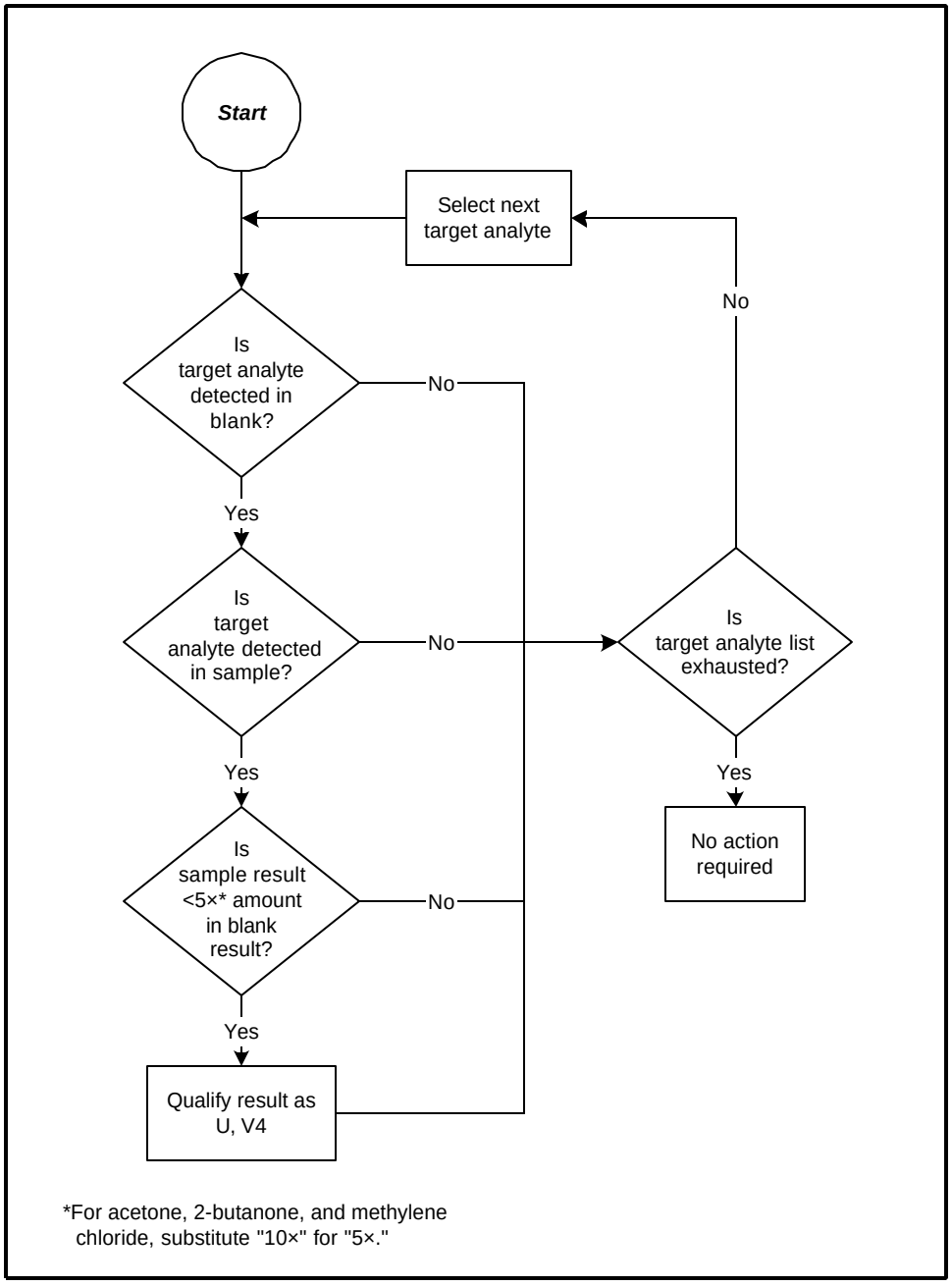

Figure 6.5-1. Applying LANL qualifier flags and reason codes to the sample results for noncompliant method blanks.

6.6 Verify Internal Standards

Note: At least three of the four approved IS compounds listed in Table 6.6-1 are *required* for each sample. Therefore, retention times and area counts *must be reported* for at least three IS compounds in all samples.

6.6.1 If *all* required IS compound (see Table 6.6-1) retention times *are reported* for all samples,

6.6.1.1 record "N" in block 1a of the VOC data validation checklist, Part IIIa;

6.6.1.2 record "n/a" in block 1c of the VOC data validation checklist, Part IIIa; and

6.6.1.3 go to Section 6.6.3.

Table 6.6-1.
ER Project-Approved VOC Internal Standards

IS Number	IS Name
1	pentafluorobenzene
2	1,4-difluorobenzene
3	chlorobenzene-d4
4	1,4-dichlorobenzene-d4

6.6.2 If the retention time for *any* of the three required IS compounds (see Table 6.6-1) is *not reported* for *any* of the requested samples,

6.6.2.1 record "Y" in block 1a of the VOC data validation checklist, Part IIIa;

6.6.2.2 circle "A, V2a" in block 1b of the VOC data validation checklist, Part IIIa;

6.6.2.3 record the qualifier flag and reason code combination "A, V2a" next to the target analyte results for which a required IS retention time was not reported, on Form I;

6.6.2.4 record, in block 1c of the VOC data validation checklist, Part IIIa, the

1) ISs for which the required retention times were not reported and

2) sample numbers to which this noncompliance applies.

6.6.3 If *all* required IS compound (see Table 6.6-1) area counts *are reported* for all samples,

6.6.3.1 record “N” in block 2a of the VOC data validation checklist, Part IIIa;

6.6.3.2 record “n/a” in block 2c of the VOC data validation checklist, Part IIIa; and

6.6.3.3 go to Section 6.6.5.

6.6.4 If the area counts for *any* of the three required IS compounds (see Table 6.6-1) *are not reported* for *any* of the requested samples,

6.6.4.1 record “Y” in block 2a of VOC validation form, Part IIIa;

6.6.4.2 circle “A, V3f” in block 2b of the VOC data validation checklist, Part IIIa;

6.6.4.3 record the qualifier flag and reason code combination “A, V3f” next to the target analyte results for which a required IS area count was not reported; and

6.6.4.4 record, in block 2c of the VOC data validation checklist, Part IIIa, the

1) ISs for which the required area counts were not reported and

2) sample numbers to which this noncompliance applies.

6.6.5 If *no* IS retention time differs by more than 30 sec from the previous continuing calibration,

6.6.5.1 record “N” in block 3a of the VOC data validation checklist, Part IIIa;

6.6.5.2 record “n/a” in block 3c of the VOC data validation checklist, Part IIIa; and

6.6.5.3 go to Section 6.6.8.

6.6.6 If *any* IS compound retention time differs by more than 30 sec from the previous calibration,

6.6.6.1 check for incorrect IS identification using retention times and mass spectra and

6.6.6.2 correct erroneous identifications.

6.6.7 For any IS retention time *verified* to differ by more than 30 sec from the previous continuing calibration,

6.6.7.1 record “Y” in block 3a of the VOC data validation checklist, Part IIIa;

6.6.7.2 circle “PM, V0” in block 3b of the VOC data validation checklist, Part IIIa;

6.6.7.3 record the qualifier flag and reason code combination “PM, V0” next to the result of each target analyte quantitated against the noncompliant IS (see the VOC data validation checklist, Part IIIb for the list of target analytes quantitated against each IS), on Form I; and

6.6.7.4 record the noncompliant ISs and the affected samples in block 3c of the VOC data validation checklist, Part IIIa.

6.6.8 If *no* IS area count is less than 10% of the previous continuing calibration area count,

6.6.8.1 record “N” in block 4a of the VOC data validation checklist, Part IIIa;

6.6.8.2 record “n/a” in block 4c of the VOC data validation checklist, Part IIIa; and

6.6.8.3 go to Section 6.6.10.

6.6.9 If *any* IS area count is less than 10% of the previous continuing calibration IS area count,

6.6.9.1 record “Y” in block 4a of the VOC data validation checklist, Part IIIa;

6.6.9.2 circle “RPM, V2” in block 4b of the VOC data validation checklist, Part IIIa;

6.6.9.3 record the qualifier flag and reason code combination “RPM, V2” next to the result of each target analyte quantitated against the noncompliant IS, on Form I; and

6.6.9.4 record the noncompliant ISs and the affected samples in block 4c of the VOC data validation checklist, Part IIIa.

6.6.10 If *no* IS area count is greater than 10% but less than 50% of the previous continuing calibration area count,

6.6.10.1 record “N” in block 5a of the VOC data validation checklist, Part IIIa;

6.6.10.2 record “n/a” in block 5c of the VOC data validation checklist, Part IIIa; and

6.6.10.3 go to Section 6.6.12.

6.6.11 If *any* IS area count is greater than 10% but less than 50% of the previous continuing calibration IS area count,

6.6.11.1 record “Y” in block 5a of the VOC data validation checklist, Part IIIa

6.6.11.2 and if any analyte quantitated against the noncompliant IS *is detected* in the sample,

1) circle “JPM, V1” in block 5b of the VOC data validation checklist, Part IIIa;

2) record the qualifier flag and reason code combination “JPM, V1” next to the result of each detected target analyte quantitated against the noncompliant IS, on Form I; and

3) record the noncompliant ISs and the affected samples in block 5c of the VOC data validation checklist, Part IIIa;

6.6.11.3 and if any analyte quantitated against the noncompliant IS *is not detected* in the sample,

1) circle “UJ, V1a” in block 5b of the VOC data validation checklist, Part IIIa;

2) record the qualifier flag and reason code combination “UJ, V1a” next to the result of each target analyte quantitated against the noncompliant IS, on Form I; and

3) record the noncompliant ISs and the affected samples, in block 5c of the VOC data validation checklist, Part IIIa.

6.6.12 If *no* IS area count is greater than 200% of the previous continuing calibration area count,

6.6.12.1 record “N” in block 6a of the VOC data validation checklist, Part IIIa;

6.6.12.2 record “n/a” in block 6c of the VOC data validation checklist, Part IIIa; and

6.6.12.3 go to Section 6.7, Verify Surrogate Recoveries.

6.6.13 If *any* IS compound area count is greater than 200% of the previous continuing calibration IS area count and a target analyte quantitated against that IS was detected,

6.6.13.1 record “Y” in block 6a of the VOC data validation checklist, Part IIIa;

6.6.13.2 circle “JPM, V1” in block 6b of the VOC data validation checklist, Part IIIa;

6.6.13.3 record the qualifier flag and reason code combination “JPM, V1” next to the result of each detected target analyte quantitated against the noncompliant IS, on Form I; and

6.6.13.4 record the noncompliant ISs and the affected samples, in block 6c of the VOC data validation checklist, Part IIIa.

6.6.14 Use the logic diagram of Figure 6.6-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant IS compounds.

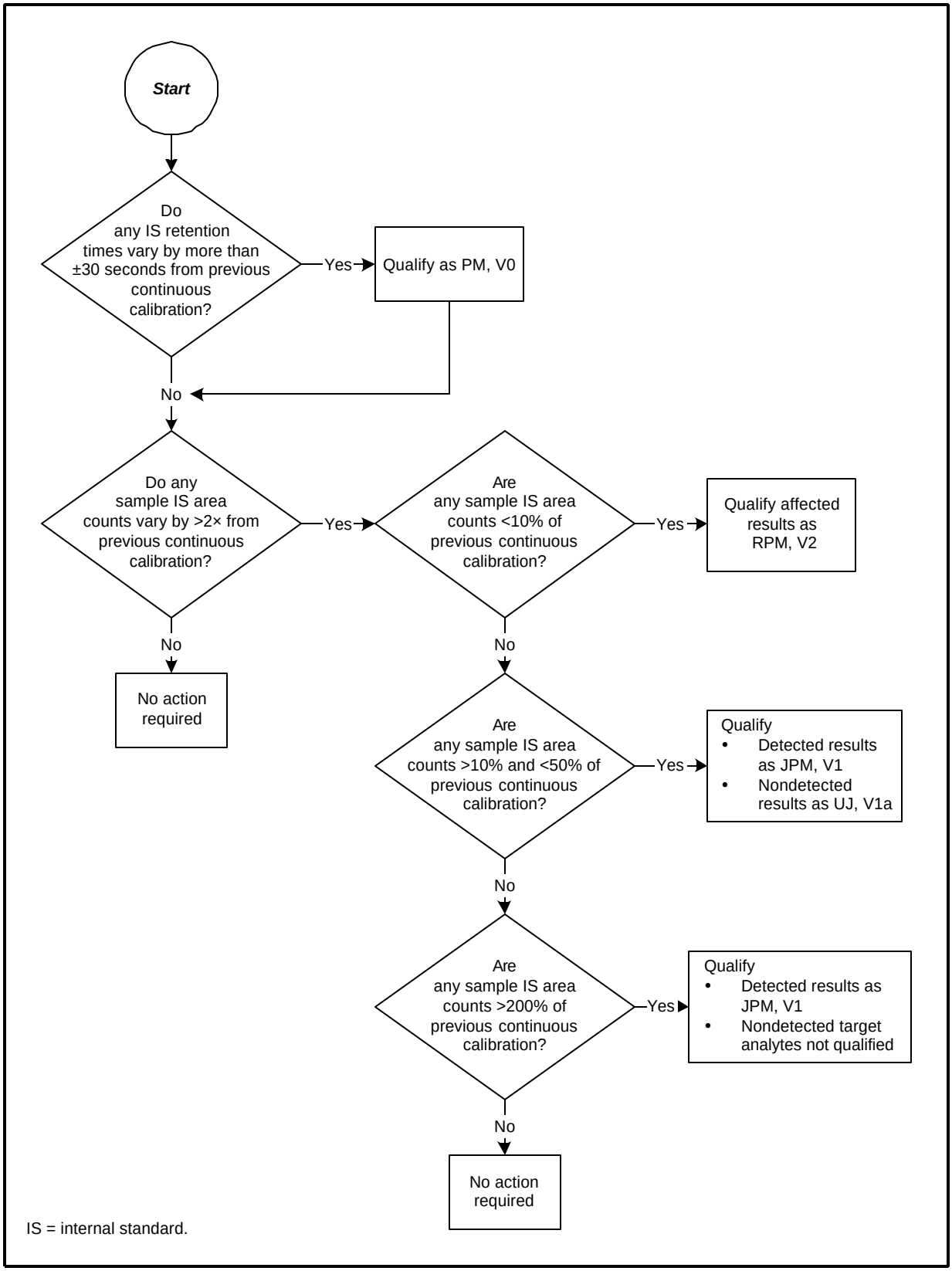

Figure 6.6-1. Applying LANL qualifier flags and reason codes to the sample results for noncompliant IS compounds.

6.7 Verify Surrogate Recoveries

Note: Surrogate %R values that are outside the acceptance range listed in Table 6.7-1 as a result of sample dilution used to render target analytes quantifiable are not subject to the validation acceptance criteria presented in this section (Section 6.7).

Table 6.7-1.
VOC Surrogates and Recovery Acceptance Ranges

Surrogate	Soil Matrix Acceptance Range (%R)	Water Matrix Acceptance Range (%R)
toluene-d8	81–117	88–110
4-bromofluorobenzene	74–121	86–115
dibromofluoromethane	80–120	86–118

6.7.1 If the %R value for *each* surrogate listed in Table 6.7-1 *is reported* for a sample,

6.7.1.1 record "N" in block 1a of the VOC data validation checklist, Part IV;

6.7.1.2 record "n/a" in block 1c of the VOC data validation checklist, Part IV; and

6.7.1.3 go to Section 6.7.3.

6.7.2 If the %R value for *any* of the surrogates listed in Table 6.7-1 is *not reported* for a sample,

6.7.2.1 record "Y" in block 1a of the VOC data validation checklist, Part IV;

6.7.2.2 circle "A, V3f" in block 1b of the VOC data validation checklist, Part IV;

6.7.2.3 record the qualifier flag and reason code combination "A, V3f" next to all results of each sample that does not contain the required surrogates; and

6.7.2.4 record the surrogate %R values that are not reported and the affected samples in block 1c of the VOC data validation checklist, Part IV.

6.7.3 For each sample, compare *all* reported surrogate %R values to the corresponding recovery acceptance range in Table 6.7-1.

6.7.4 For each sample, if *no* surrogate is less than 10%R,

6.7.4.1 record “N” in block 2a of the VOC data validation checklist, Part IV;

6.7.4.2 record “n/a” in blocks 2c and 2d of the VOC data validation checklist, Part IV; and

6.7.4.3 go to Section 6.7.6.

6.7.5 For each sample, if *at least one* reported surrogate is less than 10%R,

6.7.5.1 record “Y” in block 2a of the VOC data validation checklist, Part IV

6.7.5.2 and for target analytes that *are detected* in the affected sample,

1) circle “J-, V3b” in block 2b of the VOC data validation checklist, Part IV;

2) record the qualifier flag and reason code combination “J-, V3b” next to the result of each detected target analyte, on Form I;

3) record the noncompliant surrogates and the affected samples in block 2c of the VOC data validation checklist, Part IV; and

4) record the %R values that correspond to the identified noncompliant surrogates in block 2d of the the VOC data validation checklist, Part IV;

6.7.5.3 and for target analytes that *are not detected* in the affected sample,

1) circle “RPM, V3d” in block 2b of the VOC data validation checklist, Part IV;

2) record the qualifier flag and reason code combination “RPM, V3d” next to the result of each nondetected target analyte, on Form I;

3) record the noncompliant surrogates and the affected samples in block 2c of the VOC data validation checklist, Part IV; and

4) record the %R values that correspond to the identified noncompliant surrogates in block 2d of the VOC data validation checklist, Part IV.

6.7.6 For each sample, if *no* surrogate %R value is greater than its UAL,

6.7.6.1 record “N” in block 3a of the VOC data validation checklist, Part IV;

6.7.6.2 record “n/a” in blocks 3c and 3d of the VOC data validation checklist, Part IV; and

6.7.6.3 go to Section 6.7.10.

6.7.7 For each sample, if *at least one* surrogate %R value is greater than its UAL,

6.7.7.1 and if *no* surrogate %R values are less than their LALs,

1) record “Y” in block 3a of the VOC data validation checklist, Part IV

2) and if any target analyte *is detected* in the affected sample,

- circle “J+, V3” in block 3b of the VOC data validation checklist, Part IV;
- record qualifier flag and reason code combination “J+, V3” next to the result of each detected target analyte, on Form I;
- record the noncompliant surrogates and the affected samples, in block 3c of the VOC data validation checklist, Part IV; and
- record the %R values of the noncompliant surrogates in block 3d of the VOC data validation checklist, Part IV;

6.7.7.2 or if *at least one* surrogate %R value is less than its LAL, record

1) “N” in block 3a of the VOC data validation checklist, Part IV and

2) “n/a” in blocks 3c and 3d of the VOC data validation checklist, Part IV.

6.7.8 For each sample, if *at least one* surrogate %R value *does not* fall outside *each* acceptance limit,

6.7.8.1 record “N” in block 4a of the VOC data validation checklist, Part IV;

6.7.8.2 record “n/a” in blocks 4c and 4d of the VOC data validation checklist, Part IV; and

6.7.8.3 go to Section 6.8, Verify Holding Time.

6.7.9 For each sample, if *at least one* surrogate %R value is greater than the UAL *and* at least one surrogate %R value is less than the LAL,

6.7.9.1 record “Y” in block 4a of the VOC data validation checklist, Part IV

6.7.9.2 and if any analyte *is detected* in the affected sample,

1) circle “J, V3e” in block 4b of the VOC data validation checklist, Part IV;

2) record the qualifier flag and reason code combination “J, V3e” next to the result of each detected target analyte, on Form I;

3) record the noncompliant surrogates and the affected samples in block 4c of the VOC data validation checklist, Part IV; and

4) record the %R values that correspond to the identified noncompliant surrogates in block 4d of the VOC data validation checklist, Part IV;

6.7.9.3 and if any target analyte *is not detected* in the affected sample,

1) circle “UJ, V3e” in block 4b of the VOC data validation checklist, Part IV;

2) record the qualifier flag and reason code combination “UJ, V3e” next to the result of each undetected target analytes, on Form I;

3) record the noncompliant surrogates and the affected samples in block 4c of the VOC data validation checklist, Part IV; and

4) record the %R values that correspond to the identified noncompliant surrogates in block 4d of the VOC data validation checklist, Part IV.

6.7.10 If no surrogate is *between* its LAL and 10%R,

6.7.10.1 record “N” in block 5a of the VOC data validation checklist, Part IV;

6.7.10.2 record “n/a” in blocks 5c and 5d of the VOC data validation checklist, Part IV; and

6.7.10.3 Go to Section 6.8, Verify Holding Time.

6.7.11 If *at least one* surrogate is less than its LAL *but* greater than or equal to 10%R,

6.7.11.1 record “Y” in block 5a of the VOC data validation checklist, Part IV

6.7.11.2 and if any target analyte *is detected* in the affected sample,

1) circle “J-, V3a” in block 5b of the VOC data validation checklist, Part IV;

2) record the qualifier flag and reason code combination “J-, V3a” next to the result of each detected target analyte, on Form I;

3) record the noncompliant surrogates and the affected samples in block 5c of the VOC data validation checklist, Part IV; and

4) record the %R values that correspond to the identified noncompliant surrogates in block 5d of the VOC data validation checklist, Part IV;

6.7.11.3 and if any target analyte *is not detected* in the affected sample,

1) circle “UJ, V3c” in block 5b of the VOC data validation checklist, Part IV;

2) record the qualifier flag and reason code combination “UJ, V3c” next to the result of each undetected target analyte, on Form I;

3) record the noncompliant surrogates and the affected samples in block 5c of the VOC data validation checklist, Part IV; and

4) record the %R values that correspond to the identified noncompliant surrogates in block 5d of the VOC data validation checklist, Part IV.

6.7.12 Use the logic diagram of Figure 6.7-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant surrogate compounds.

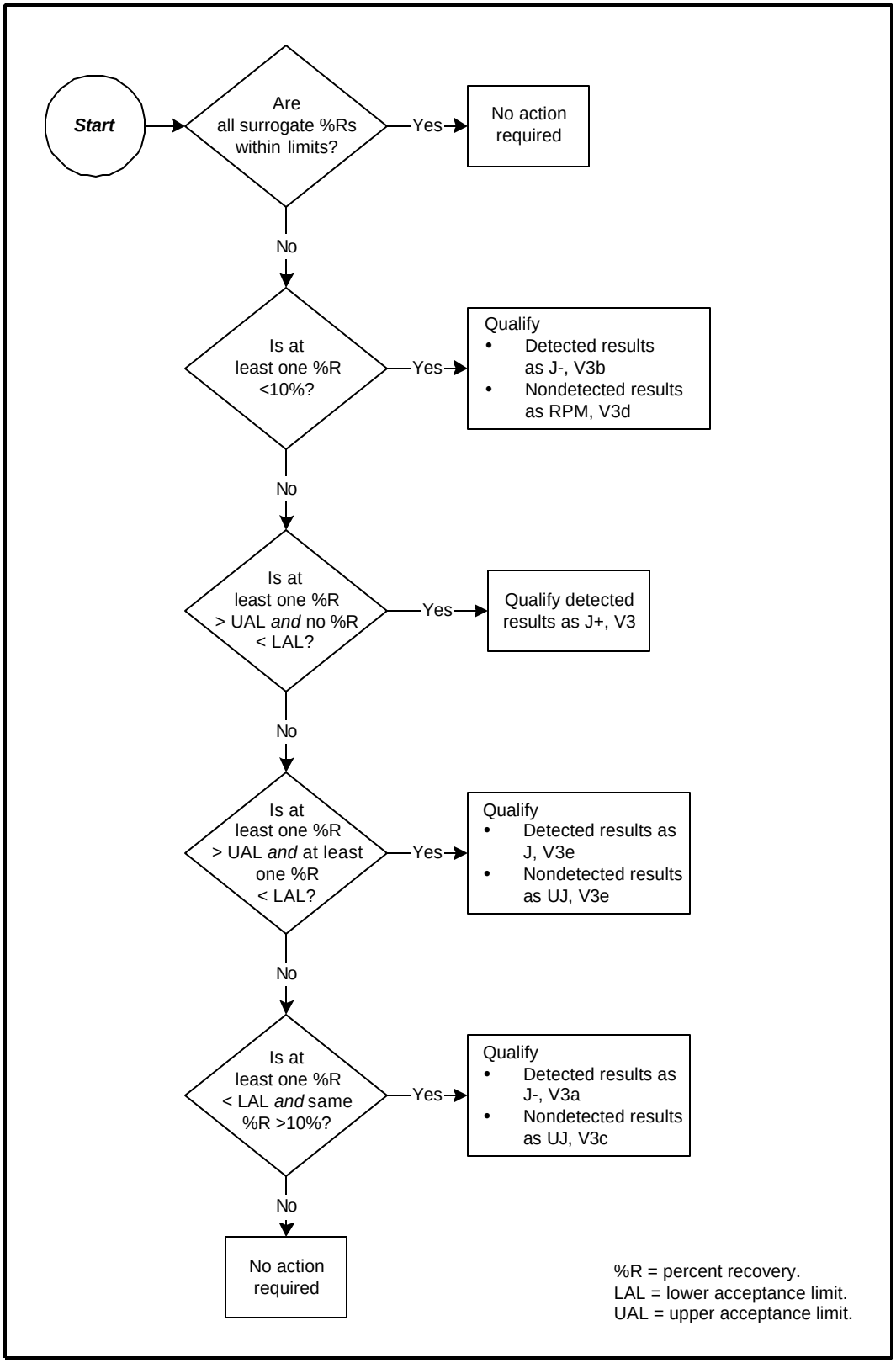

Figure 6.7-1. Applying LANL qualifier flags and reason codes to the sample results for noncompliant surrogate compounds.

6.8 Verify Holding Time

6.8.1 If *all* samples *were analyzed* within their holding times (see Table 6.8-1),

6.8.1.1 record "Y" in block 1a of the VOC data validation checklist, Part V;

6.8.1.2 record "n/a" in blocks 1c and 1d of the VOC data validation checklist, Part V; and

6.8.1.3 go to Section 6.9, Verify Tentatively Identified Compounds.

Table 6.8-1.
Holding Time Acceptance Criteria*

Sample Matrix	Recommended Holding Time (days)
Soil	14
Water	7 if not acidified; 14 if acidified
*Applicable storage conditions are listed in the current SOW for analytical services.	

6.8.2 If *any* samples *were not analyzed* within the holding time (see Table 6.8-1),

6.8.2.1 record "N" in block 1a of the VOC data validation checklist, Part V;

6.8.2.2 circle "PM, V9" in block 1b of the VOC data validation checklist, Part V;

6.8.2.3 record the qualifier flag and reason code combination "PM, V9" next to the result of each affected target analyte, on Form I;

6.8.2.4 record which samples are affected in block 1c of the VOC data validation checklist, Part V; and

6.8.2.5 record (for each sample that exceeded holding time) the number of days by which the holding time was exceeded in block 1d of the VOC data validation checklist, Part V.

6.9 Verify Tentatively Identified Compounds

Note: If the order code contains an "N" as the last letter, the "N" indicates that TICs were *not* requested.

6.9.1 If the ER Project *did not request* TIC reporting,

6.9.1.1 record "N" in block 1a of the VOC data validation checklist, Part VI;

6.9.1.2 record “n/a” in block 1c of the VOC data validation checklist, Part VI; and

6.9.1.3 go to Section 6.10, Complete the Data Validation Cover Sheet.

6.9.2 If the ER Project *requested* TIC reports (i.e., “N” does not follow the order code on the COC form),

6.9.2.1 and if TICs *are not present* in *any* samples,

1) record “n/a” in blocks 1a and 1c of the VOC validation form, Part VI and

2) go to Section 6.10, Complete the Data Validation Cover Sheet;

6.9.2.2 if TICs *are present* in the sample and TIC forms *are not available*,

3) record “Y” in block 1a of the VOC data validation checklist, Part VI;

4) circle “A, V11” in block 1b of the VOC data validation checklist, Part VI;

5) record the qualifier flag and reason code combination “A, V11” to each sample for which TIC forms are not available, on Form I; and

6) record the samples for which TIC forms are not available in block 1c of the VOC data validation checklist, Part VI.

6.9.3 If TICs *are present* and *are reported* in at least one sample, record

6.9.3.1 “N” in block 1a of the VOC data validation checklist, Part VI and

6.9.3.2 the samples that contain TICs, in block 1d of the VOC data validation checklist, Part VI.

6.10 Complete the data validation cover sheet by signing and dating it.

6.11 Assemble the validation data record package to include the following items in the order they are listed below:

6.11.1 the completed, signed, and dated data validation cover sheet;

6.11.2 the VOC data validation checklists completed in Sections 6.2 through 6.9;

6.11.3 photocopies of the form (Form I) on which data validation qualifiers and reason codes have been recorded (assemble in order by sample identification);

6.11.4 a photocopy of the data record package case narrative; and

6.11.5 photocopies of the data record package QC forms.

6.12 Submit the validation data record package to the SMO in accordance with ER-SOP-15.09.

7.0 REFERENCES

The following documents are cited within this procedure:

EPA (US Environmental Protection Agency), February 1994. “US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review,” Publication 9240.1-05, EPA-540/R-94/012, Office of Solid Waste and Emergency Response, Washington, DC.

ER-SOP-15.09, Chain of Custody for Analytical Data Packages

LANL (Los Alamos National Laboratory), July 1995. “Environmental Restoration Project Statement of Work for Analytical Services,” Revision 2, RFP Number 9-SX1-Q4257, Los Alamos National Laboratory, Los Alamos, New Mexico.

QP-2.2, Personnel Orientation and Training

QP-4.2, Standard Operating Procedure Development

8.0 RECORDS

Although no records will be submitted to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 6.11 will be a part of the data record package submitted to the RPF from the SMO in accordance with ER-SOP-15.09.

9.0 ATTACHMENTS

Users of this document may employ documentation formats different from those attached to/named in this procedure *if* the substituted formats provide, as a minimum, the information required in the official forms developed by this procedure. This procedure includes the following attachments:

Attachment A: Volatile Organic Data Validation Qualifier Flags (1 page)

Attachment B: Volatile Organic Data Validation Reason Codes (2 pages)

Attachment C: Data Validation Cover Sheet (1 page)

Attachment D: Volatile Organic Data Validation Checklist forms (9 pages)

Volatile Organic Data Validation Qualifier Flags

A	The contractually required supporting documentation for this datum is absent.
U	The analyte is classified as “not detected.”
J	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual.
J+	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.
J-	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.
UJ	The analyte is classified as “not detected” with an expectation that the reported result is more uncertain than usual.
RPM	The reported sample result is classified as “rejected” due to serious noncompliance regarding quality control acceptance criteria. The presence or absence of the analyte cannot be verified based on routine validation alone.
PM	Manual review of raw data is recommended to determine if the observed noncompliances with quality acceptance criteria adversely impacts data use.

Note: A “PM” qualifier flag indicates that a manual review should be conducted if the datum that is qualified with the “PM” is important to the data user. In addition, "PM" also means that a decision must be made by the project manager/delegee regarding the need for further review of the data. This review should include some consideration of potential impact that could result from using the “PM” qualified data.

Volatile Organic Data Validation Reason Codes

V0 The IS retention time has shifted by more than ±30 seconds, which could affect compound identification and cause false positives or negatives to be reported.

V1 The IS area count for the quantitating IS is outside the -50%–+100% window in relation to the previous continuing calibration. This condition could affect the quantitation accuracy of the associated analytes.

V1a The area count for the quantitating IS is less than 50% of the area count for the previous continuing calibration, greatly increasing the potential for false negative results.

V2 The quantitating IS area is less than 10% of the expected value, which indicates an increased potential for false negative results and possibly other problems with sample quantitation.

V2a Required IS information is missing. Data may not be acceptable for use.

V3 The surrogate percent recovery is greater than the UAL, which indicates the potential for a high bias in the results and the potential for false positive results.

V3a The surrogate is less than the LAL but greater than or equal to 10%R, which indicates the potential for a low bias in the results.

V3b The surrogate is less than 10%R and the result is a detect, which indicates the potential for a severely low bias in the results.

V3c The quantitation acceptance limit is approximated for nondetects because a surrogate is less than the LAL but greater than or equal to 10%R, which indicates a significant potential for false negative results.

V3d The surrogate is less than 10%R and the result is a nondetect, which indicates a greatly increased potential for false negative results.

V3e At least one surrogate is greater than the UAL and one surrogate is less than the LAL, which indicates a greater than normal degree of uncertainty in the result.

V3f Required surrogate information is missing. Data may not be acceptable for use.

V4 The sample result is greater than the EQL and less than or equal to 5 times (10 times for acetone, methylene chloride, and 2-butanone) the concentration of the related analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.

V5a Method-blank data is missing, or method blank was not analyzed. Data may not be acceptable for use.

V9 The holding time is exceeded. The data user should evaluate the data of interest with respect to the effects of exceeding the holding time. Factors to consider include sample preservation, sample storage practices, use of the data, levels of contamination found in the sample, and the physical, chemical, and biological stability of the target analytes in the sample matrix.

V11 TICs are not reported by the analytical laboratory but were requested by the ER Project. The analytical laboratory was contacted and TICs were not provided.

V14a Insufficient sample volume was received for a matrix spike and/or a matrix spike duplicate analysis.

Note: A matrix spike duplicate is not appropriate for all analyses.

V14b The matrix spike and/or the matrix spike duplicate analysis was not performed on a sample associated with a LANL request number.

Note: A matrix spike duplicate is not appropriate for all analyses.

V14c The matrix spike and/or the matrix spike duplicate was analyzed on a sample associated with a different LANL request number but no summary was included.

Note: A matrix spike duplicate is not appropriate for all analyses.

V15 Because the sample was damaged, lost, or of insufficient quantity, the laboratory was unable to analyze it.

V16 Required calibration information is missing or samples were analyzed on an expired calibration. Data may not be acceptable for use.

Data Validation Cover Sheet

Section I.

Request Number:_____ Validation Date:_____ Lab Code:_____

Contract Laboratory Name: _____

Validator:_____ Organization:_____

Analytical Suite (check all that apply): ☐ Volatile Organics ☐ Semivolatile Organics ☐ Organochlorine Pesticides/Polychlorinated Biphenyls ☑ High Explosives ☐ Inorganics ☐ Radiochemistry

Other (describe): _____

Section II. Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
☐	☐	☐	1. Chain-of-custody form(s)	☐	☐	☐	6. Raw/BSS data
☐	☐	☐	2. Case narrative	☐	☐	☐	7. Quality control forms
☐	☐	☐	3. Sample result forms	☐	☐	☐	8. Quantitation reports
☐	☐	☐	4. Sample chromatograms	☐	☐	☐	9. TICs forms
☐	☐	☐	5. Standard chromatograms	☐	☐	☐	10. TIC mass spectra

Identify any samples in the assigned Request Number that are missing:

_____ _____ _____ _____ _____ _____ _____ _____

_____ _____ _____ _____ _____ _____ _____ _____

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Validator's signature: _____ Date: _____

ER-SOP-15.01	Los Alamos Environmental Restoration Project

Volatile Organic Data Validation Checklist

Part I. Instrument Performance Checks and Calibrations

Criteria	Criterion true? (y, n, or n/a)	Action if "criterion true?" = no Assign qualifier …	Actual time lapse
Was the instrument performance check completed on the same date as, or within 12 hours of, the corresponding sample analyses?	1a.	1b. "**A, V16**" for all samples without acceptable calibration. In block 1c, record the actual time lapse.	1c.
Was the initial calibration completed within 12 hours of completing the instrument performance check?	2a.	2b. "**A, V16**" for all samples without acceptable calibration. In block 2c, record the actual time lapse.	2c.
Was the continuing calibration check performed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and before the analysis of blanks and samples? **Attention:** A continuing calibration check is not required if all samples are analyzed within 12 hours of initial calibration—record "n/a" in blocks 3a and 3c.	3a.	3b. "**A, V16**" for all samples without acceptable calibration. In block 3c, record the actual time lapse.	3c.
ER-SOP-15.01		Los Alamos Environmental Restoration Project	

Volatile Organic Data Validation Checklist

Part IIa. Method Blanks Validation Criteria

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier …	List affected matrices or batches.
Was a method blank analyzed for each sample matrix and/or 12-hour batch beginning with the BFB injection?	1a.	1b. "**A, V5a**" for any missing documentation. In block 1c, record all sample matrices and/or analytical batches that did not include a method blank.	1c.

Part IIb. Method Blanks Validation Criteria (continued)

Criteria	Criterion true? (yes or no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List detected blank analyte(s) and affected samples.	Analyte concentration (mg/kg)
Is a target analyte detected in both the method blank and sample **AND** is the sample result = 5 times the method-blank concentration*?	2a.	2b. "**U, V4**" to the sample analyte(s) in question (in blocks 2c and 2d).	2c.	2d.

* Replace "5 times" with "10 times" for laboratory contaminants acetone, 2-butanone, or methylene chloride.

Volatile Organic Data Validation Checklist

Part IIIa. Internal Standard (IS) Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List all noncompliant ISs, samples, retention times, & area counts.
Are any required IS retention times *not* reported?	1a.	1b. "**A, V2a**" for any missing documentation. In block 1c, record • any ISs not reported and • all affected samples.	1c.
Are any required IS area counts *not* reported?	2a.	2b. "**A, V2a**" for any missing documentation. In block 2c, record • any ISs not reported and • all affected samples.	2c.
Is any sample IS retention time >30 seconds different from previous continuing calibration?	3a.	3b. "**PM, V0**" to all sample analytes quantitated against the IS in question.	3c.
Are any sample IS area counts <10% of previous continuing calibration IS area counts?	4a.	4b. "**RPM, V2**" to all detected sample analytes quantitated against the IS in question.	4c.
Are any sample IS area counts >10% **AND** <50% of previous continuing calibration area counts?	5a.	5b. "**JPM, V1**" to *detected* sample analytes quantitated against the IS in question. **"UJ, V1a**" to *nondetected* sample analytes quantitated against the IS in question.	5c.
Is a target analyte detected in sample **AND** any sample IS area counts >200% of previous continuing calibration's IS area counts?	6a.	6b. "**JPM, V1**" to detected sample analytes quantitated against the IS in question.	6c.

Volatile Organic Data Validation Checklist

Part IIIb. Internal Standard Assignments

1. Pentafluorobenzene	2. 1,4-Difluorobenzene	3. Chlorobenzene-d4	4. 1,4-Dichlorobenzene-d4
Acetone	Benzene	Bromoform	Bromobenzene
Bromochloromethane	Bromodichloromethane	Chlorobenzene	n-Butylbenzene
Bromomethane	4-Bromofluorobenzene (*surrogate*)	Chlorodibromomethane	sec-Butylbenzene
2-Butanone	Carbon tetrachloride	1,3-Dichloropropane	tert-Butylbenzene
Carbon disulfide	1,2-Dibromoethane	Ethylbenzene	2-Chlorotoluene
Chloroethane	Dibromomethane	2-Hexanone	4-Chlorotoluene
Chloroform	1,2-Dichloroethane	Styrene	1,2-Dibromo-3-chloropropane
Chloromethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	1,2-Dichlorobenzene
Dichlorodifluoromethane	1,1-Dichloropropene	Tetrachloroethene	1,3-Dichlorobenzene
1,1-Dichloroethane	c-1,3-Dichloropropene	o,m,p-Xylene (mixed isomers)	1,4-Dichlorobenzene
1,1-Dichloroethene	t-1,3-Dichloropropene		Isopropylbenzene
c-1,2-Dichloroethene	1,1,2-Trichloroethane		p-Isopropyltoluene
t-1,2-Dichloroethene	Toluene		n-Propylbenzene
2,2-Dichloropropane	Toluene-d8 (*surrogate*)		1,1,2,2,-Tetrachloroethane
Iodomethane	Trichloroethene		1,2,3-Trichloropropane
Methylene chloride	4-Methyl-2-pentanone		1,2,4-Trimethylbenzene
1,1,1-Trichloroethane			1,3,5-Trimethylbenzene
Trichlorofluoromethane			
Vinyl chloride			

The following internal standard assignments will vary: Dibromofluoromethane (surrogate) and Trichlorotrifluoroethane

Volatile Organic Data Validation Checklist

Required VOC Surrogate Compounds and Recovery Acceptance Ranges

Surrogate name	Soil matrix recovery acceptance range	Water matrix recovery acceptance range
Toluene-d8	81%–117%	88%–110%
4-Bromofluorobenzene	74%–121%	86%–115%
Dibromofluoromethane	80%–120%	86%–118%

ER-SOP-15.01

Los Alamos
Environmental Restoration Project

Volatile Organic Data Validation Checklist

Part IV. Surrogate Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Identify noncompliant surrogates *and* assign qualifier & reason code…	List all noncompliant surrogates and samples.	Percent recovery
Are any required surrogate percent recoveries *not* reported?	1a.	1b. "**A, V3f**" for any missing documentation. In block 1c, record • noncompliant surrogates and • all affected samples.	1c.	1d. n/a
Is at least one surrogate recovery <10%?	2a.	2b. "**J-, V3b**" to all *detected* sample analytes and "**RPM, V3d**" to all *nondetected* sample analytes.	2c.	2d.
Is at least one surrogate percent recovery > UAL **AND** no surrogate percent recoveries < LAL?	3a.	3b. "**J+, V3**" to all detected sample analytes.	3c.	3d.
Is at least one surrogate percent recovery > UAL **AND** at least one surrogate percent recovery <LAL?	4a.	4b. "**J, V3e**" to all *detected* sample analytes and "**UJ, V3e**" to all *nondetected* sample analytes.	4c.	4d.
Is at least one surrogate percent recovery < LAL **AND** *the same* surrogate percent recovery ≥ UAL?	5a.	5b. "**J-, V3a**" to all *detected* sample analytes and "**UJ, V3c**" to all *nondetected* sample analytes.	5c.	5d.

Volatile Organic Data Validation Checklist

Part V. Holding Time Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = no Assign qualifier & reason code…	List samples for which holding times were exceeded	List the number of days by which holding times were exceeded.
Was each sample analyzed within its required holding time?	1a.	1b. "**PM, V9**" to all analytes in affected samples.	1c.	1d.

ER-SOP-15.01

Los Alamos
Environmental Restoration Project

Volatile Organic Data Validation Checklist

Part VI. Tentatively Identified Compounds (TICs) Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = no Assign qualifier & reason code…	Samples that are missing TICs forms	Samples that contain TICs
Were TICs requested (i.e., an "**N**" is *not* appended to the analysis order code on the chain of custody form) **AND** TICs were *not* reported in at least one sample?	1a.	1b. "**A, V11**" to samples that are missing documentation. In block 1d, record the sample numbers of each sample for which TICs have been reported.	1c.	1d.